IN THE UNITED STATES PATENT AND TRADEMARK OFFICE
BEFORE THE BOARD OF PATENT APPEALS AND INTERFERENCES

In re the Application of:   ONO, Toshiaki et al.                     Group Art Unit:   1765

Serial No.:   09/883,922                              Examiner:   ANDERSON, Matthew A.

Filed:   June 20, 2001                                  Confirmation No.:   3735

For:   METHOD OF MANUFACTURING EPITAXIAL WAFER AND METHOD OF
PRODUCING SINGLE CRYSTAL AS MATERIAL THEREFOR

Attorney Docket No.:   010789
Customer Number:   38834

SUBMISSION OF APPEAL BRIEF

Commissioner for Patents
P.O. Box 1450
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**MAR 0 8 2004**

Date:   March 3, 2004

Sir:

Submitted herewith are an original and two copies of an Appeal Brief in the
above-identified U.S. patent application.

Also enclosed is a check in the amount of $ 440.00.   This check includes $330.00 to
cover the cost of filing this Appeal Brief and $ 110.00 for one-month extension of time.   In the
event that any additional fees are due with respect to this paper, please charge Deposit Account
No. 50-2866.   This paper is filed in triplicate.

Respectfully submitted,

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APPEAL BRIEF FOR THE APPELLANT

MAR 0 8 2004

Ex parte   Toshiaki ONO, et al. (applicants)

## METHOD OF MANUFACTURING EPITAXIAL WAFER AND METHOD OF PRODUCING SINGLE CRYSTAL AS MATERIAL THEREFOR

Serial Number:   **09/883,922**

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Date:   March 3, 2004

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## APPEAL BRIEF

This is an appeal from the Office Action dated September 11, 2003 in which claims 1, 2, 4, 5, 8-11 and 13-16 were finally rejected.

A Notice of Appeal was filed on December 11, 2003.

## I. REAL PARTY IN INTEREST

The real party in interest is **Sumitomo Mitsubishi Silicon Corporation**, by assignment recorded in the U.S. Patent and Trademark Office on **July 5, 2002**, at Reel **01360** / Frame **0349**.

## II. RELATED APPEALS AND INTERFERENCES

Appellants know of no other appeals or interference proceedings related to the present appeal.

## III. STATUS OF CLAIMS

Claims 1, 2, 4, 5, 8-11 and 13-16 on appeal have been finally rejected under 35 USC §103(a) as obvious over the prior art. Claim 15 is also rejected under 35 USC §112, second paragraph as being indefinite. Claims 3, 6, 7 and 12 have been canceled.

## IV. STATUS OF AMENDMENTS

An amendment after final is filed concurrently herewith to address rejection under 35 USC §112.

## V. CLAIMS ON APPEAL

A clean copy of claims 1, 2, 4, 5, 8-11 and 13-16 on appeal is attached hereto as an Appendix. The clean copy of claims incorporates the proposed amendments after final rejection for the purposes of the appeal.

## VI. SUMMARY OF THE INVENTION

The present invention relates to a method of manufacturing a silicon single crystal by the Czochralski method and to a method of manufacturing an epitaxial wafer from the silicon crystal produced by that method. More particularly, it relates to a method of manufacturing an epitaxial wafer exhibiting high gettering capability while scarcely giving rise to stacking faults, dislocations and other defects in an epitaxial layer (epitaxial layer defects) when it is grown on a wafer obtained from a silicon single crystal produced with or without doping with nitrogen, and to a method of producing such a single crystal to serve as a raw material for the epitaxial wafer. An object of the present invention is to provide a method of manufacturing epitaxial wafers by which the thermal stability of oxide precipitates is retained even after the epitaxial step without requiring any additional heat treatment step following silicon single crystal pulling up and thus epitaxial wafers capable of exhibiting high IG capability are provided.

Another object of the present invention is to provide a method of manufacturing epitaxial wafers which solves the problem concerning epitaxial defects resulting from doping with nitrogen and which provides epitaxial wafers allowing formation of a sufficient amount of oxide precipitates upon high temperature heat treatment in the device step while preventing the generation of epitaxial layer defects even when they are manufactured from a silicon single crystal grown with nitrogen doping.

## 1.   The first method of manufacturing epitaxial wafers (without nitrogen doping)

The first single crystal is produced by selecting a cooling rate of not less than 7.3°C/min within the temperature range of 1200-1050°C in crystal pulling up in the Czochralski method and according to the method of manufacturing epitaxial wafers according to the present invention, and an epitaxial layer is grown on the surface of silicon wafers sliced from that single crystal.

By rapidly cooling at a rate of not less than 7.3°C/min within the temperature range of 1200-1050°C during the first single crystal pulling up, vacancies introduced at the solid-liquid interface can be prevented from aggregation or cohesion and thus the density of residual vacancies can be maintained at a high level.   As a result, the free energy for the formation of oxide precipitate nuclei decreases and the growth of oxide precipitate nuclei starts within a higher temperature range than in the prior art.   Therefore, the thermal stability of oxide precipitate nuclei increases and, thus, a sufficient number of oxide precipitates can be formed even in the heat treatment in the device process following epitaxial growth.

Therefore, according to the method of manufacturing epitaxial wafers which uses the first single crystal, the effects of IG can fully be produced from early stages of the device step by causing an epitaxial layer on the surface of silicon wafers sliced from the silicon single crystal produced by controlling the thermal history thereof, as mentioned above, without any additional heat treatment step prior to the epitaxial step.

The second single crystal is produced by the Czochralski method by selecting a cooling rate of not less than 7.3°C/min in the temperature range of 1200-1050°C in the step of pulling up and a cooling rate of not more than 3.5°C/min in the temperature range of 1000-700°C.   The epitaxial layer is grown on the surface of silicon wafers sliced from that single crystal.

The rapid cooling in the temperature range of 1200-1050°C in the first stage of the cooling step to be controlled in pulling up the second single crystal produces the same effects as the cooling of the first single crystal. Further, the subsequent slow cooling in the temperature range of 1000-700°C in the next stage is based on the finding that the oxide precipitate nuclei formed can be grown and rendered more thermally stable by such slow cooling.

Within the temperature range of 1000-700°C, a slow rate of cooling of not more than 3.5°C/min is employed such that the cooling rate produces sufficient effects of slow cooling to stably increase the density of oxide precipitates.

Even in the method of manufacturing epitaxial wafers which uses the second single crystal, when an epitaxial layer is grown on the surface of silicon wafers sliced from the silicon single crystal produced by controlling the thermal history in the manner mentioned above, the oxide precipitate nuclei will not shrink or vanish upon epitaxial treatment without needing any additional heat treatment step, like in the production method which uses the first single crystal.

The oxygen content in the silicon wafers sliced from the first or second single crystal is made not less than $12 \times 10^{17}$ atoms/cm$^3$ (ASTM '79). Silicon single crystals produced by the Czochralski method generally contain about $10^{18}$ atoms/cm$^3$ of supersaturation oxygen but, when the oxygen content is insufficient, a marked decrease in wafer strength may result and/or sufficient IG effects will not be produced in certain instances. Therefore, it is desirable that the oxygen concentration be not less than $12 \times 10^{17}$ atoms/cm$^3$ (ASTM '79) so that the stability of oxide precipitate nuclei can be effectively secured when the first or second single crystal is used.

## 2.   The second method of manufacturing epitaxial wafers (with nitrogen doping)

The third single crystal is made by a method characterized in that, in pulling up a silicon single crystal doped with $1 \times 10^{12}$ atoms/cm$^3$ to $1 \times 10^{14}$ atoms/cm$^3$ of nitrogen in the

Czochralski method, a cooling rate of not less than 2.7°C/min is employed in the single crystal temperature range of 1150-1020°C.

In the third single crystal, the dopant nitrogen concentration is restricted to $1 \times 10^{12}$ atoms/cm³ to $1 \times 10^{14}$ atoms/cm³ because the effect of thermally stabilizing oxide precipitate nuclei is low, hence those oxide precipitate nuclei which occur within the wafer inside disappear upon high temperature heat treatment in the epitaxial step, when the nitrogen concentration is lower than $1 \times 10^{12}$ atoms/cm³, while when the concentration is higher than $1 \times 10^{14}$ atoms/cm³, the effect of thermally stabilizing oxide precipitate nuclei is conversely excessive, hence those oxide precipitate nuclei having high thermal stability and occurring in the vicinity of the wafer surface will not vanish even when subjected to high temperature heat treatment in the epitaxial step but rather cause generation of epitaxial layer defects.

The temperature range for the above cooling is restricted to 1150-1020°C since the etch pit density after heat treatment can increase and the oxide precipitate density can increase accordingly when rapid cooling is carried out in that temperature range. Further, the rate of cooling is restricted to 2.7°C/min or more since this is a cooling rate found securable and appropriate in the above pulling rate changing test mentioned above; it has been confirmed that sufficient cooling effects can be produced at such a rate.

Thus, in the method of producing the third single crystal, the nitrogen concentration is restricted to a relatively low level within the range of $1 \times 10^{12}$ atoms/cm³ to $1 \times 10^{14}$ atoms/cm³ to thereby suppress the formation of thermally stable oxide precipitate nuclei and, on the other hand, the single crystal is rapidly cooled in the temperature range of 1150-1020°C to thereby prevent vacancies incorporated at the solid-liquid interface from cohesion and cause the formation of oxide precipitate nuclei on the higher temperature side and thus allow oxide

precipitate nuclei to grow. By this, it is possible to prevent epitaxial layer defects from being generated and attain sufficient oxide precipitate formation even in the heat treatment after epitaxial growth.

In accordance with the second epitaxial wafer manufacturing method, an epitaxial layer is grown on the surface of silicon wafers sliced from the third silicon single crystal. By this, it is possible to manufacture epitaxial wafers high in IG capability with scarce occurrence of epitaxial layer defects. In other words, it is possible to manufacture high-quality epitaxial wafers free of defects in the device active region.

The method of producing the fourth single crystal is characterized in that, in pulling up the silicon single crystal doped with $1 \times 10^{12}$ atoms/cm$^3$ to $1 \times 10^{14}$ atoms/cm$^3$ in the Czochralski method, a cooling rate of not more than 1.2°C/min is employed in the single crystal temperature range of 1000-850°C.

Here, slow cooling is employed in the temperature range of 1000-850°C because, as is evident from the results shown in Fig. 2, the growth of oxide precipitate nuclei can be promoted even by slow cooling in that temperature range and thereby oxide precipitate nuclei relatively large in size can be formed. Based on the results of the above-mentioned pulling rate changing test, a slow cooling rate of not more than 1.2°C/min is employed as the cooling rate in the above restricted temperature range; sufficient oxide precipitate nuclei growth can be attained at such a cooling rate.

In accordance with the second epitaxial wafer manufacturing method, an epitaxial layer is grown on the surface of silicon wafers sliced from the fourth silicon single crystal, whereby epitaxial wafers exhibiting high IG capability can be manufactured with scarce occurrence of epitaxial layer defects.

### 3. The fifth single crystal and the method of manufacturing epitaxial wafers using the same

The method of producing the fifth single crystal is characterized in that, in pulling up the silicon single crystal doped with $1 \times 10^{12}$ atoms/cm$^3$ to $1 \times 10^{14}$ atoms/cm$^3$ of nitrogen in the Czochralski method, a cooling rate of not less than 2.7°C/min is employed in the single crystal temperature range of 1150-1020°C and, thereafter, a cooling rate of not more than 1.2°C/min is employed in the temperature range of 1000-850°C.

The fifth single crystal can be produced by combining the method of producing the third single crystal with the method of producing the fourth single crystal.

In accordance with the second epitaxial wafer manufacturing method, an epitaxial layer is grown on the surface of silicon wafers sliced from the fifth silicon single crystal, whereby epitaxial wafers exhibiting high IG effects can be produced with scarce occurrence of epitaxial defects, as in the case where the third or fourth single crystal is used.

The sixth single crystal is produced by a method which is characterized in that, in pulling up the silicon single crystal doped with $5 \times 10^{13}$ atoms/cm$^3$ to $1 \times 10^{16}$ atoms/cm$^3$ of nitrogen in the Czochralski method, a cooling rate of not less than 6.5°C/min is employed in the single crystal temperature range of 1150-800°C.

From the viewpoint of preventing epitaxial layer defects from occurring, it is effective to select a low nitrogen concentration, as mentioned hereinabove. However, an increased nitrogen concentration thermally stabilizes those oxide precipitate nuclei which occur within the wafer and prevents them from vanishing upon high temperature heat treatment to thereby contribute toward the increase in oxide precipitate density, hence it is effective to utilize such increased nitrogen concentration.

The present inventors conjectured that even thermally stable oxide precipitate nuclei occurring near the wafer surface, if not large in size, would vanish upon high temperature heat treatment for epitaxial growth and have now arrived at the method of producing the sixth single crystal.

For the sixth single crystal, a relatively high nitrogen concentration is selected within the dopant nitrogen concentration range of $5 \times 10^{13}$ atoms/cm$^3$ to $1 \times 10^{16}$ atoms/cm$^3$ to cause the formation of thermally stable oxide precipitate nuclei. And, by rapidly cooling at a rate of not less than 6.5°C/min in the temperature range of 1150-800°C, namely by cooling as rapidly as possible in the temperature range of 1150-800°C, which corresponds to the temperature range in which void defects are formed (cf. Fig. 3) and the temperature range in which oxide precipitate nuclei are grown (cf. Fig. 4), it is intended that the formation of void defects be supposedly causative of epitaxial defects and the growth of thermally stable oxide precipitate nuclei be both prevented.

The upper limit to the nitrogen concentration for doping the sixth single crystal is set at 1 $\times 10^{16}$ atoms/cm$^3$ because, at a level exceeding this limit, single crystals have dislocations in single crystal growing and the product yield thus markedly decreases. The lower limit to the cooling rate is 6.5°C/min (rapid cooling) since, at a lower rate, the effect of preventing voids from cohesion is slight and the effect of suppressing the growth of oxide precipitate nuclei is also slight due to the high dopant nitrogen concentration, with the result that thermally stable oxide precipitate nuclei large in size are formed.

In accordance with the second epitaxial wafer manufacturing method, an epitaxial layer is grown on the surface of silicon wafers sliced from the sixth silicon single crystal, whereby epitaxial wafers exhibiting high IG capability can be manufactured with scarce occurrence of

epitaxial layer defects, as in the case where one of the third to fifth single crystals is used. In other words, it is possible to manufacture high-quality epitaxial wafers free of defects in the device active region.

In the second epitaxial wafer manufacturing method, it is desirable that the oxygen content in the above-mentioned third to sixth single crystals be not less than $4 \times 10^{17}$ atoms/cm$^3$ (ASTM '79) so that a sufficient amount of oxide precipitates to produce sufficient IG effects can be secured. The reason is that while the silicon single crystals produced by the Czochralski method generally contain about $10^{18}$ atoms/cm$^3$ of supersaturation oxygen, as mentioned above, an insufficient oxygen concentration results in decreases in wafer strength and/or failure to produce satisfactory IG effects.

The method of doping to be employed in the second epitaxial wafer manufacturing method may be any one provided that doping with a predetermined concentration of nitrogen can be realized.

## VII. ISSUES

Whether the invention, as recited in claims 1, 2, 4, 5, 8-11 and 13-16 on appeal is obvious over Iida et al and Wolf et al under 35 USC §103(a).

## VIII. GROUPING OF THE CLAIMS

The rejected claims 1, 2, 4 and 5 on appeal stand or fall together in that they recite "a cooling rate of not less than 7.3 °C/min in the single crystal temperature range of 1200-1050°C," and that "the single crystal has an oxygen concentration of not less than $12 \times 10^{17}$ atoms/cm$^3$ (ASTM '79 value), and the single crystal is not nitrogen doped."

The rejected claims 8, 9, 13 and 14 on appeal stand or fall together in that they recite "a silicon single crystal doped with $1 \times 10^{12}$ atoms/cm$^3$ to $1 \times 10^{14}$ atoms/cm$^3$ of nitrogen in the Czochralski method," "cooling . . . a cooling rate of not more than 1.2°C/min in the single crystal temperature range of 1000-850°C," and "wherein the single crystal has an oxygen concentration of not less than $12 \times 10^{17}$ atoms/cm$^3$ (ASTM '79 value)."

The rejected claims 10 and 15 on appeal stand or fall together in that they recite that the silicon crystal is "doped with $5 \times 10^{13}$ atoms/cm$^3$ to $1 \times 10^{16}$ atoms/cm$^3$ of nitrogen" in the Czochralski method "with a cooling rate of not less than 6.5°C/min in the single crystal temperature range of 1150-800°C" and that "the single crystal has an oxygen concentration of not less than $12 \times 10^{17}$ atoms/cm$^3$ (ASTM '79 value)."

## IX. ARGUMENT WITH RESPECT TO ISSUES

### A.   THE REFERENCES

The Examiner relied upon two prior art references, namely, Iida et al (EP0962557) and Wolf et al (Silicon Processing For The VLSI Era, Volume 1: Process Technology, Lattice Press, Sunset Beach, CA, USA, pp. 8-11, 27-33, 36-72, 124, 139-142, 1986) in the 35 USC §103(a) rejection of claims 1, 2, 4, 5 and 8-16 on appeal.

Iida et al discloses a silicon single crystal wafer and method for producing the silicon single crystal wafer. Iida et al describes as follows:

> [0010] As described above, in the silicon single crystal wafer produced by processing a silicon single crystal ingot grown by CZ method with doping nitrogen, grown-in defects present on the wafer is very few due to presence of doped nitrogen. When the crystal ingot is grown with controlling the rate of cooling from 1150 °C to 1080 °C at 2.3 °C/min or more, the size of the crystal defects can be extremely decreased to have almost no harmful influence on fabrication of the device. Furthermore, since growth of crystal defects can be suppressed, it is possible to grow crystal at high speed, so that productivity can also be improved.
>
> [0011] In the above case, the nitrogen concentration in the above-mentioned silicon single crystal wafer is preferably in the range of 0.2 to 5 x $10^{15}$ atoms/cm$^3$.
>
> [0012] In order to suppress growth of crystal defect, the nitrogen concentration is preferably 1 x $10^{10}$ atoms/cm$^3$ or more. In order not to prevent crystallization of single crystal, the nitrogen concentration is preferably 5 x $10^{15}$ atoms/cm$^3$ or less. However, the nitrogen concentration in the range of 0.2 to 5 x $10^{15}$ atoms/cm$^3$ is the most effective for suppression of growth of crystal defects, and therefore growth of crystal defects can be sufficiently suppressed when the nitrogen concentration is in the range.

[0013] During growth of the silicon single crystal ingot by Czochralski method with doping nitrogen, a concentration of oxygen in the single crystal ingot is preferably controlled to be $1.0 \times 10^{18}$ atoms/cm$^3$ (ASTM '79 value) or less. When the oxygen concentration is low as above, growth of the crystal defects can be further suppressed, and formation of oxide precipitates in the surface layer can be prevented.

(paragraph [0010] to [0013]). The Examiner noted on the disclosure as follows:

Iida et al. discloses a method of pulling a silicon single crystal ingot according to the Cz (Czochralski) method. The cooling rate of the ingot from 11 50-1080°C was disclosed as 2.30°C/min or more. The rapid cooling was said to decrease the density and size of the grown in defects. Nitrogen doping was also suggested in the range from $1.0 \times 10^{10}$ to $5 \times 10^{15}$ atoms/cm$^3$ (page 2 item 12). Paragraph 10 page 2 indicates that a faster crystal growth speed (i.e. a faster cooling rate) improves productivity. Iida discloses an oxygen concentration of $1.0 \times 10^{18}$ atoms /cm$^3$ or less in item 13 on page 2. This value prevents surface defects although it is silent concerning bulk wafer defects.

Iida et al. does not specify the cooling rate in the 1000-850°C temperature range.

Wolf et al discloses various aspects of silicon processing for the VLSI area. Wolf et al page 11 was referred to for allegedly disclosing that interstitial agglomeration defects are prevented by quenching during the temperature range where interstitial point defects are mobile within the crystal lattice (i.e. from 1421°C to about 950°C). The cited portion on page 11 describes as follows:

The dislocation-free attribute of CZ crystals refers to macroscopic (edge and screw) dislocations. Small dislocation loops, however, can be formed during the growth of the main crystal by the condensation of excess point defects (Chap. 2). This phenomenon occurs as the crystal cools from the solidification temperature (1421°C) down to about 950°C. During this cooldown, the solubility of the point defects created at temperatures near meltdown is reduced, until the crystal is supersaturated with point defects. If these point defects can diffuse sufficiently, they will

tend to arrive at sites at which they can agglomerate and thereby be removed from solution. If the pull-rate is high enough, the solidified region cools with sufficient rapidity that the excess point defects are immobilized (or *quenched*) in the lattice. They thereby have insufficient opportunity to diffuse and agglomerate into clusters large enough to cause dislocation loops. Both interstitial silicon and oxygen have been suggested as the point defect species responsible for forming such agglomerates. For crystals in which the pull-rate has not been sufficiently high, the presence of the resultant dislocation loops will cause the crystal to exhibit *swirl* patterns when wafers are subjected to defect-delineation chemical etching. Swirl has also been linked to the growth-induced defects stemming from fluctuations occurring at the [continued to p.12] . . . .

Wolf et al page 27 was cited for allegedly suggesting an oxygen concentration of 25 ppma. The Examiner appears to refer to Table 2.

Wolf et al page 32 was cited for allegedly disclosing nitrogen doping for the same purpose (it is not clear what it the same purpose).

. . . [continuing from page 31] walls to the solidification interface is reduced, and less oxygen is incorporated into the ingot (7 ppma for MCZ, versus 13-19 ppma for conventional CZ silicon). With lower oxygen content, significantly less oxygen thermal donor formation is observed, and oxygen-associated defects (precipitates and stacking faults) are reported to be suppressed. MCZ is being investigated by several silicon manufacturers (Fig. 25). *Controlled evaporation of the dopant from the melt* has reportedly been successfully utilized to grow an antimony-doped CZ silicon ingot with uniform axial doping. Antimony (Sb) shows a much higher evaporation rate than P, B, or As, and if it can be evaporated in a controlled manner to keep the melt concentration of Sb constant (in spite of its small segregation coefficient $k_0$=0.023 ) a more uniformly doped ingot will result.

The *incorporation of nitrogen* in CZ and FZ silicon is being studied as a technique to increase the warpage resistance of wafers. Oxygen is known to increase wafer mechanical strength, and nitrogen appears to play a similar role. Unlike oxygen, which can outdiffuse during thermal processing (and thereby cause the wafer surface region strength to be reduced), the surface [continuing to

page 32] concentration of nitrogen is much more constant due to its small diffusion coefficient in Si. The intrinsic gettering properties of nitrogen (Chap. 2) are as yet unknown.

Wolf et al page 49 was cited for allegedly disclosing that the temperature range of the diffusion (i.e. mobility) of supersaturated Si self-interstitial point defects is between about 400 to 1200°C. Wolf et al describes as follows:

The rate of formation of precipitates depends on the temperature, degree of supersaturation, and time of cooling (or annealing). Each point defect requires a certain time, depending on its diffusivity, to reach the precipitate. Thus, faster diffusing point defects may form the first precipitates, even though slower diffusing point defects may also be present in a crystal at higher levels of supersaturation. Once precipitation occurs, and nuclei of one point-defect type are formed, further precipitation by condensation at the first nuclei can take place more easily for all types of point defects14.

The mechanism of precipitation that occurs in a freshly-grown CZ crystal as it cools to room temperature provides a useful example of the general mechanism of precipitation. In such a cooling crystal, there are more point defects (self-interstitials, vacancies, and impurities) than are soluble at the lowered temperature. This creates a departure from equilibrium as the crystal cools, and establishes a driving force that invites precipitation. Specifically, while the interior of the ingot is still hot (~400-1200°C), any fast-diffusing supersaturated point defects (e.g. oxygen or self-interstitials) can still move over significant distances in the crystal. Precipitates can nucleate on extended crystal defects, such as dislocations and stacking faults. In the days prior to availability of dislocation-free silicon, point-defect precipitation and excess point defect annihilation occurred primarily on such grown-in dislocations. However, if there are no extended crystal defects that can act as nucleation centers, as in dislocation-free CZ silicon crystals, the point-defect type which first exceeds the solubility limit during cooling is then able to form precipitation nuclei with its own species *(homogeneous nucleation)*. These types of nuclei (Fig. I ib) are sometimes referred to as *point-defect clusters.*

Wolf et al pages 59-61 have been cited for allegedly showing that oxygen in Si is detailed as forming precipitates at approximately 550-800°C. The Examiner also noted that the processing parameters are result effective as disclosed on page 61.

### OXYGEN IN SILICON

Other than in heavily doped crystals, the most abundant impurity in Czochralski (CZ) Si crystal is oxygen, with concentrations typically ranging from $5 \times 10^{17}$- $1 \times 10^{18}$ atoms/cm$^3$ (or 10-20 ppma). The majority of the oxygen (~95%) in the as-grown crystal is atomically dissolved, and occupies interstitial sites. The diffusivity of oxygen is moderately high (Fig. 19), whereas its solubility decreases strongly at lower temperatures (Fig. 2). These two properties make oxygen the most important precipitate-forming element in CZ-silicon. The precipitation behavior affects all of the important effects influenced by the presence of oxygen in silicon including: a) donor formation; b) surface oxygen precipitates; c)bulk oxygen precipitates (that provide the basis of intrinsic gettering); and d) wafer warpage resistance 32.

The mechanism by which oxygen is incorporated into CZ silicon was described in Chap. 1, and the kinetics of precipitate formation was introduced in the *Bulk Defects* section. The characterization methods to measure oxygen concentration levels in silicon were presented sections dealing with *FTIR* in Chaps. 1 and 5. In this section, and the one following on *Gettering*, we complete the precipitation formation discussion as it applies to oxygen in silicon, and thereby also cover thermal donor formation.

Oxygen is incorporated as a result of crucible erosion during.CZ growth. In general, higher concentrations of oxygen are found at the seed end of the ingot because more crucible wall area is in contact with the melt when the crucible is full. In addition, the convection currents which contribute to the crucible erosion rate, are much stronger in a full crucible than at later stages of growth when the crucible is emptier.

Oxygen in silicon is able to form up to $3 \times 10^{16}$ donors fcm3 in the temperature range of 300-500°C (highest formation rate is at ~450°C), and this is an undesirable property from the viewpoint of manufacturing devices. Apparently, oxygen moves from interstitial locations to [page 60 starts here] substitutional sites and

thus forms a donor effect. More recent investigations show that a second type of donor can also be formed at temperatures of *550-800°C*, although these tend to form more slowly. *Low-temperature oxygen donors* arise from the formation of oxygen-silicon complexes, $SiOx$, and these can be annihilated by 650°C heat treatment (Fig. 20). The growth of *high-temperature oxygen donors* seems to involve carbon, since carbon at levels of ~1 ppma is found in the crystals in which they form. The higher-temperature donors are destroyed by heat treatments at 900°C. None of the existing donor formation models have been able to closely confirm the experimentally observed relationships. A review of the proposed models of the structure of oxygen donors, and the diffusivity of oxygen in Si at temperatures between 300-400°C can be found in Ref. 56. Whatever the exact model for these donors, they are significant because considerable "back end" processing of MOS devices occurs near 450°C (e.g. during passivation, wire bonding, and packaging processes). In some cases, degradation of device parametric performance has been attributed to low-temperature donor formation.

The standard 650°C heat treatment to eliminate low temperature donors is limited by the inability to quickly quench large wafers, and the possibility that high-temperature donors may be forming during the anneal. In addition, the normal 1-2 hour 600-700°C anneal can cause the formation of very small oxygen precipitates that allow more rapid oxygen precipitation at higher [page 61 starts here] temperatures. Short RTP cycles, on the order of seconds at 650-750°C, have been suggested as an effective alternative annealing step.

In silicon which contains oxygen at levels <6 ppma, so few oxygen donors are formed that their presence is not measurable. Thus, another way to effectively eliminate the effects of oxygen donors *is.* to form a denuded zone at the wafer surface, and to create oxygen precipitation in the wafer bulk. This will reduce the interstitial oxygen content in both the surface and the bulk to levels below the minimum required to form oxygen donors.

As described in Chap. 1, the time/temperature conditions endured by different parts of the ingot can vary substantially. For example, the seed-end of the ingot undergoes a long exposure to 400°C, while at the tang end this temperature exposure is short. Thus, wafers that indicate identical measurable levels of interstitial oxygen, may in fact contain different actual oxygen concentrations, since some of the oxygen may be present in precipitate (rather than

in interstitial) form. As a result, the subsequent precipitation behavior of the two wafers may be substantially different. Therefore, starting wafers will need to be supplied in a form that will exhibit predictable and reproducible precipitation kinetics at specific oxygen concentrations and temperature cycles. This is especially true if intrinsic gettering is to be used. It has been demonstrated that only reproducible precipitation will lead to reliable intrinsic gettering. The precipitation behavior can be inferred from measurements of the interstitial oxygen content before and after a high heat treatment (e.g. 1050°C for 24 h).

The presence of surface oxygen precipitates adversely impacts the quality of thin gate oxides and nucleates oxygen-induced stacking faults, which can cause device failures by excessive leakage currents, etc. It would be useful if these were not present at the surface. In addition, if the precipitates are to be used to getter unwanted impurities from the active device regions, it is necessary that the precipitates be prohibited from forming in such regions. The procedure utilized to prevent oxygen precipitates from forming near the wafer surface involves creation of a *denuded* zone, or zone that contains less interstitial oxygen than required to form oxygen precipitates. The thermal steps used to create denuded zones are outlined in the section that describes gettering.

It was mentioned in Chap. 1, and again earlier in this chapter, that the presence of oxygen enhances the warpage resistance of silicon. However, it has been reported that if high oxygen content and high temperature cycles occur simultaneously, very large (~2000A) precipitates can be formed. These are surrounded by large dislocations, which can grow and move through the lattice under the presence of strain, and result in warpage. On the other hand, silicon wafers in which the oxygen is interstitially dispersed, or forms small clusters (50A), appear to strengthen and cause wafers to exhibit a higher elastic limit. Figure 16 shows the comparative values of the critical shear stress in silicon at different temperatures and varying precipitate concentrations[22]. The critical shear stress increases with both reduced wafer temperature and reduced oxygen precipitate formation. However, such results also appear to be dependent on detailed material and experimental process conditions.

Wolf et al page 66 has been cited for allegedly disclosing that oxygen precipitates are good for intrinsic gettering sites. The Examiner referred to Fig. 16 (not shown here) for allegedly disclosing the relationship of oxide in Si to wafer warpage resistance.

### Intrinsic Gettering

*Intrinsic gettering* is based on the principle that under proper conditions, supersaturated oxygen in silicon wafers will precipitate out of solution, and form clusters within the wafer during thermal processing. The stresses that result as these clusters grow into larger precipitates can be relieved by punching out dislocation loops (or by emitting Si-self interstitials, that agglomerate to form stacking faults and dislocation loops). These dislocations become sites at which unwanted impurities can be trapped and localized'. Than effectively designed intrinsic gettering process, these precipitates are only allowed to form in the bulk regions of the wafer. They are prevented from forming in the active device regions by reducing the oxygen concentrations to levels below the threshold required for precipitation (denuded zone formation). In this manner, unwanted impurities are localized. (gettered) only in regions not containing active devices (Fig. 22). The advantages of intrinsic over extrinsic gettering include the following: a) the technique may be employed without any external treatment of the wafer other than heating; b) the volume of the wafer provides a sink during intrinsic gettering that is, roughly two orders of magnitude larger than the volume of the gettering region on the wafer backside; and c) the gettering region in the bulk is much closer to the device regions than the backside of the wafer36, so that the distance required for impurities to diffuse from the circuit regions to the sinks is 25-50 times shorter. Therefore, intrinsic gettering allows diffusion cycles which are significantly shorter than those required for extrinsic gettering to remove the same contamination

The first requirement **for** successfully implementing this technique is that the starting wafers must have appropriate oxygen concentration levels (i.e. 15-19 ppma). The lower limit of this range is somewhat above the threshold concentration of oxygen required for precipitation [continuing to page 67] ($\sim 6 \times 10^{17}$ atoms/cm$^3$=12 ppma).

Wolf et al page 140 has been cited for allegedly disclosing the warpage resistance as critical for epitaxial Si wafers. Wolf et al page 141 has been cited for allegedly disclosing the use of IG (intrinsic gettering) of the substrates prior to deposition.

### Defects Induced During Epitaxial Deposition and Their Nucleation Mechanisms

The most common types of defects that occur as a result of epitaxial deposition are dislocations and stacking faults. Dislocations are generated in epitaxial films by several mechanisms. These include: a) the propagation into the growing film of a dislocation line in the substrate that reaches the substrate surface. (Heavily dislocated substrates result in heavily dislocated films. Therefore, dislocation-free substrate material should be used); b) the existence of a large difference in lattice parameter between the film and the substrate (e.g. when the substrate is very heavily doped and the epi layer lightly doped), resulting in *misfit dislocations.* The mismatch is relieved by forming misfit dislocations in the epitaxial film near the substrate; and c) by thermally generated stresses that exceed the yield strength of the silicon, resulting in slip.

Thermal stresses are set up by temperature gradients that exist between the back and front of the wafers during the deposition process. These gradients cause a sequence of events that ultimately lead to slip generation. This sequence of events is as follows (and is worse for if than for IR heating of the sUSCeptors used for epitaxial depositions): As the if heats the sUSCeptor (which in turn heats the back of the wafers by radiation and conduction from the sUSCeptor), the front sides of the wafers radiate heat to the environment. This results in a temperature difference of several degrees between the back and front of the wafers, and to different amounts of thermal expansion between the front and back. Since the back surface of the wafer expands more than the front surface, the wafer assumes a bowl-like shape to accommodate this effect (wafer bow). This causes the wafer edges to lift from the sUSCeptor. The wafer edges now receive less heat from the sUSCeptor, and cool to a lower temperature than the remainder of the wafer, causing even further wafer bow. The bowing eventually results in stress levels that exceed the yield strength of the silicon at the elevated temperature, resulting crystal slip. This effect is depicted in Fig. 17. The temperature difference tends to increase

for larger diameter wafers. The problem can substantially be reduced in if heated reactors by shaping the sample holders into pockets, so the wafers make contact with the sUSCeptor only on the edges (see Fig. 18). By placing heat reflectors above the wafers, the temperature difference between the back and front surface can also be reduced. A recent solution to the slip problem includes the use of a radiant heater focused on the front side of the wafers to reduce the net heat flux through the wafers. In IR heated reactors, on the other hand, heating from the front side is already provided, which tends to minimize the problem.

The nucleation of *stacking faults in* the epitaxial layer is believed to result from two distinct causes: a) microscopic surface steps on the substrate; and b) impurities either on the substrate or within the reactor itself. As presented earlier, an atomistic model for the growth of an epitaxial film has been developed. In this model, the film tends to propagate by two-dimensional steps lying along the <110> directions. Ravi has shown that these side-moving steps may encounter an obstacle on the substrate, resulting in a fault in the stacking sequence (see Chap. 2). The presence of slip bands in the substrate, or particles on the substrate can also nucleate stacking faults. Local regions of silicon dioxide, nitride, or carbide on the surface also provide sites for nucleation of such faults. Sources of these compounds include: a) residual amounts of moisture or carbon dioxide in the reactor (which may grow $SiO2$ on the substrate) during the deposition; and b) hydrocarbon contamination, which can result in pockets of $SiC$ on the substrate. Metallic impurities ($Fe$, $Cu$, $Ni$) can become incorporated in the vapor phase and subsequently be deposited with the silicon film as a solid solution. As a result of the rapid cooling from the deposition temperature, these impurities may remain in solid solution even at room temperature (quenching). Further heat treatment, such as a subsequent oxidation, can cause these impurities to precipitate, resulting in oxidation-induced stacking faults.

Particulates within the reactor that land on the substrate can also serve as sites for stacking fault nucleation. Radiantly heated barrel reactors have n effective boundary layer (flow is parallel to the wafer surface), which protects the substrates from particle injection from the gas phase. Particle transport across this boundary layer can be further reduced by using low lamp power, which eliminates electrostatic migration of particles.

**B.  THE REJECTION UNDER 35 USC §103(a)**

The Examiner first rejected claims 8-16 on appeal as follows:

> 2. Claims 8-16 are rejected under 35 USC. 103(a), as being
> unpatentable over Iida et al (EP0962557) in view of Wolf et al.
> (Silicon Processing for the VLSI Era, Volume 1: Process
> Technology, Lattice Press, Sunset Beach, CA, USA, pp. 8-11,
> 27-33, 36-72, 124, 139-142, 1986.).
>    Iida et al. discloses a method of pulling a silicon single
> crystal ingot according to the Cz (Czochralski) method. The
> cooling rate of the ingot from 1150-1080°C was disclosed as
> 2.30°C/min or more. The rapid cooling was said to decrease the
> density and size of the grown in defects. Nitrogen doping was also
> suggested in the range from 1.0 x $10^{10}$ to 5 x $10^{15}$ atoms/cm 3
> (page 2 item 12). Paragraph 10 indicates that a faster crystal
> growth speed (i.e. a faster cooling rate) improves productivity. Iida
> discloses an oxygen concentration of 1.0 x $10^{18}$ atoms /cm3 or less
> in item 13 on page 2. This value prevents surface defects although
> it is silent concerning bulk wafer defects.
>    Iida et al. does not specify the cooling rate in the
> 1000-850°C temperature range.
>
>    Wolf et al. on page 11 discloses that interstitial
> agglomeration defects are prevented by quenching during the
> temperature range interstitial point defects are mobile within the
> crystal lattice (i.e. from 1421°C to about 950°C). The
> agglomerated interstitial point intrinsic defects are dislocation
> loops. On page 49, it is further intimated that the temperature range
> of the diffusion (i.e. mobility) of supersaturated Si self- interstitial
> point defects is between about 400 to 1200 degrees Celsius. On
> pages 59- 61 oxygen in Si is detailed as forming precipitates at
> approximately 550-800°C. On page 66, oxygen precipitates are
> disclosed as good for intrinsic gettering sites. Fig. 16 discloses the
> relationship of oxide in Si to wafer warpage resistance. Nitrogen
> doping for the same purpose is suggested on page 32. Page 140
> discloses the warpage resistance as critical for epitaxial Si wafers.
> On page 141 the use of IG (intrinsic gettering) of the substrates
> prior to deposition is described. The examiner notes that the
> processing parameters are result effective as disclosed on page 61.
>
>    It would have been obvious to one of ordinary skill in the
> art at the time of the present invention to combine Iida et al. with
> Wolf et al. because Iida et al. discloses the quenching of Si ingots
> pulled with the Cz method to prevent the agglomeration of vacancy

point defects in a certain temperature range and Wolf et al. discloses that the quenching in the temperature range of 1421 to 400 °C prevents agglomeration of interstitial and vacancy point defects in Cz Si. The use of quench cooling in Cz Si pulling is thus well established.

In respect to claims 8, 9, 10, 13, 14, 15 it would have been to one of ordinary skill in the art at the time of the present invention to optimize the cooling rate because Iida et al. suggests faster rates increase the productivity of the process and such optimization would have been achieved with only routine experimentation.

Further, it would have been obvious to one of ordinary skill in the art at the time of the present invention to optimize the temperature range of fast cooling because Iida et at. discloses such ranges as useful for Si defect control and Wolf et at. suggests defect agglomeration as reduced with quenching (i.e. high cooling rates).

In respect to claims 8, 9, 10, 11, 13, 14, 15, 16, it would have been obvious to one of ordinary skill in the art at the time of the present invention to optimize the oxygen and nitrogen concentration of the Si single crystal because Wolf et al suggests an oxygen concentration of 25 ppma (page 27; this is 1.25 xl 018 atoms/cm$^3$ by extrapolation of the typical concentrations given on page 59) and suggests nitrogen doping for warpage resistance. Oxygen content was known to allow oxygen precipitates and thus IC to form in the silicon wafer.

In respect to claims 13, 14, 15, it would have been obvious to one of ordinary skill in the art at the time of the present invention to grow an epitaxial layer on a silicon wafer produced from an Si ingot because such epitaxial layers (including those formed on IG substrate wafers) were known in the art. (see Wolf page 124).

In respect to claims 8, 9, 10, 13, 14, 15 it would have been obvious to one of ordinary skill in the art at the time of the present invention to optimize the fast cooling and slow cooling of a silicon crystal through the specified temperature ranges because this was disclosed as useful for forming oxygen precipitates and thus IC substrates for further production of Silicon epitaxial wafers and because such optimization of result effective process parameter would have been achieved with only routine experimentation.

Then the Examiner rejected claims 1, 2, 4 and 5 as follows:

> 3. Claims 1, 2, 4, 5 are rejected under 35 USC. 103(a) as being unpatentable over Iida et al (EP0909840) in view of Wolf et al. (Silicon Processing for the VLSI Era, Volume 1: Process Technology, Lattice Press, Sunset Beach, CA, USA, pp. 8-11, 27-33, 36-72, 124, 139-142, 1986.).
>
> Iida et al. discloses a method of pulling a silicon single crystal ingot according to the Cz (Czochralski) method. The cooling rate of the ingot from 1150-1080°C was disclosed as 20 minutes or less (i.e. a cooling rate of 3.5°C/minute or more). The cooling from 1250-1200°C was also given as at this rate in an alternative embodiment. The rapid cooling was said to decrease the density and size of the grown in defects. The rapid cooling was described to give an excellent chip yield (abstract). No disclosure of nitrogen doping is given in Iida et al.
>
> Iida et al. does not Iida et al. does not [sic] specify the cooling rate in the 1200-1050°C temperature range be 7.3°C/min or more.
>
> Wolf et al. on page 11 discloses that interstitial agglomeration defects are prevented by quenching during the temperature range interstitial point defects are mobile within the crystal lattice (i.e. from 1421 °C to about 950°C). The agglomerated interstitial point intrinsic defects are dislocation loops. On page 49, it is further intimated that the temperature range of the diffusion (i.e. mobility) of supersaturated Si self interstitial point defects is between about 400 to 1200 degrees Celsius. On pages 59- 61 oxygen in Si is detailed as forming precipitates at approximately 550-800°C. On page 66, oxygen precipitates is disclosed as good for intrinsic gettering sites. Fig. 16 discloses the relationship of oxide in Si to wafer warpage resistance. Wolf et al suggests an oxygen concentration of 25 ppma (page 27); this is 1.25 xl 018 atoms/cm3 by extrapolation of the typical concentrations given on page 59) as typical for Cz pulled Si single crystal. Page 140 discloses the warpage resistance as critical for epitaxial Si wafers. On page 141 the use of IG (intrinsic gettering) of the substrates prior to deposition is described. The examiner notes that the processing parameters are result effective as disclosed on page 61.
>
> It would have been obvious to one of ordinary skill in the art at the time of the present invention to combine Iida et al. with

Wolf et al. because Iida et al. discloses the quenching of Si ingots pulled with the Cz method to prevent the agglomeration of defects in a certain temperature range and Wolf et al. discloses that the quenching in the temperature range of 1421 to 400 °C prevents agglomeration of interstitial and vacancy point defects in Cz Si. The use of quench cooling in Cz Si pulling is thus well established.

In respect to claims 1, 2, 4, 5, it would have been to one of ordinary skill in the art at the time of the present invention to optimize the cooling rate because Iida et al. suggests faster rates increase the productivity of the process and such optimization would have been achieved with only routine experimentation.

Further, it would have been obvious to one of ordinary skill in the art at the time of the present invention to optimize the temperature range of fast cooling because Iida et al. discloses such ranges as useful for Si defect control and Wolf et al. suggests defect agglomeration as reduced with quenching (i.e. high cooling rates).

In respect to claims 1, 2, 4, 5, it would have been obvious to one of ordinary skill in the art at the time of the present invention to optimize the oxygen concentration of the Si single crystal because and Wolf et al suggests an oxygen concentration of 25 ppma (page 27; this is 1.25 xl 018 atoms/cm3 by extrapolation of the typical concentrations given on page 59) and suggests oxygen as important to reducing defects in epitaxial wafers by the formation of IG oxygen precipitates (page 141 -1 42).

In respect to claim 4, 5, it would have been obvious to one of ordinary skill in the art at the time of the present invention to grow an epitaxial layer on a silicon wafer produced from an Si ingot because such epitaxial layers (including those formed on IG substrate wafers) were known in the art. (see Wolf page 124).

In respect to claims 1, 2, 4, 5, it would have been obvious to one of ordinary skill in the art at the time of the present invention to optimize the fast cooling and slow cooling of a silicon crystal through the specified temperature ranges because this was disclosed as useful for forming oxygen precipitates and thus IG substrates for further production of Silicon epitaxial wafers and because such optimization of result effective process parameters would have been achieved with only routine experimentation.

The Examiner commented on Applicants' previous argument as follows:

> 4. Applicant's arguments filed 6/20/2003 have been fully considered but they are not persuasive.
>
> The argument that the combination does not suggest the epitaxial IG wafers produced by the present method is not convincing. The examiner points to the known use of temperature and cooling control to effect both the limitation of defect formation and the promotion of beneficial defect formation (i.e. the oxygen precipitates vital to Intrinsic Gettering). Wolf et al. describes IG as beneficial, thus at least suggests a sufficient amount of oxygen precipitates was previously obtained.
>
> The teaching away argument is not convincing. Iida (EP 0990840) discloses few crystal defects without specifying the oxygen concentration required. Iida (EP 0962557) merely states a preference for 'low' oxygen to add to the defect avoidance. This is not rule out higher concentrations. Page 67 of Wolf et al. suggests optimization of oxygen concentrations for Si wafers.
>
> The argument that neither Iida [n]or Wolf suggests a method of increasing the density of oxygen precipitates is not convincing. Wolf et al. discloses oxygen precipitate creation on page 61. This suggests an increase in density. Page 67 discloses a method of nucleation and growth of oxygen precipitates.
>
> The argument against the formation of precipitates in the range from 550-800°C is not convincing. Page 67 Fig. 24 shows nucleation (i.e. formation) of oxygen precipitates from donors at 600°C.
>
> The arguments directed to claims 1, 2, 4, 5 are moot in light of the new grounds of rejection necessitated by amendment.
>
> The argument that the specific cooling rates and ranges are not in the references is not persuasive. The references in combination suggest the result effective parameter optimization of the cooling rate and temperature ranges.

Claim 15 has been rejected under 35 USC 112 as follows:

5. Claim 15 is rejected under 35 USC. 112, second paragraph, as being indefinite for failing to particularly point out and distinctly claim the subject matter which applicant regards as the invention.

The phrase "...with $5 \times 10^{13}$ atoms/cm$^3$ to $1 \times 10^{16}$ atoms/cm$^3$. . ." is not particular as to what the dopant is or is not.

From other claims in the application, the examiner has deduced this is to refer to nitrogen doping and has examined the claim above under this assumption.

### C.    APPELLANTS' ARGUMENT

**1.    Rejection Under 35 USC §112, Second Paragraph Has been Overcome By The Amendment After Final.**

In item 5 of the Office Action, claim 15 is rejected under 35 USC §112, second paragraph, as being indefinite because the phrase "… with $5 \times 10^{13}$ atoms/cm³ to $1 \times 10^{16}$ atoms/cm³ …" is not particular as to what the dopant is or is not.

Accordingly, an amendment after final has been filed to overcome the rejection.

**2.    The Examiner has not established prima facie case of Obviousness.    Also, claims 8-16 are not obvious over <u>Iida et al</u> (EP0962557) in view of <u>Wolf et al</u>.**

Claims 8-16 are rejected under 35 USC. 103(a), as being unpatentable over <u>Iida et al</u> (EP0962557) in view of <u>Wolf et al</u>.    Claims 8, 9, 13 and 14 recite, among other things, "cooling the silicon single crystal with **a cooling rate of not more than 1.2°C/min in the single crystal temperature range of 1000-850°C,**" and "wherein the single crystal has **an oxygen concentration of not less than $12 \times 10^{17}$ atoms/cm³** (ASTM '79 value)."

The Examiner alleged, at page 2, last line of the Office Action, that "Iida et al. does not specify the cooling rate in the 1000-850 °C temperature range," implying that all the other recitations are disclosed in <u>Iida et al</u>.    <u>Iida et al</u>, however, also fails to disclose "wherein the single crystal has **an oxygen concentration of not less than $12 \times 10^{17}$ atoms/cm³** (ASTM '79 value)."    The Examiner further cited <u>Wolf et al</u> and alleged the obviousness, on page 3 of the Office Action, as follows:

Wolf et al. **on page 11** discloses that interstitial agglomeration defects are prevented by quenching during the temperature range interstitial point defects are mobile within the crystal lattice (i.e. from 1421°C to about 950°C). **On page 49,** it is further intimated that the temperature range of the diffusion (i.e. mobility) of supersaturated Si self-interstitial point defects is between about 400 to 1200 degrees Celsius. **On page 59-61,** oxygen precipitates are disclosed as good for intrinsic gettering sites. Fig. 16 [on page 54] discloses the relationship of oxide in Si to wafer warpage resistance. Nitrogen doping for the same purpose is suggested **on page 32.** **Page 140** discloses the warpage resistance as critical for epitaxial Si wafers. **On page 141** the use of IG (intrinsic gettering) of the substrates prior to deposition is described. The examiner notes that the processing parameters are result effective as disclosed **on page 61.**

It would have been obvious . . . to combine Iida et al. with Wolf et al. because Iida et al. discloses the quenching of Si ingots pulled with the Cz method to prevent the agglomeration of vacancy point defects in a certain temperature range and Wolf et al. discloses that the quenching in the temperature range of 1421 to 400 °C prevents agglomeration of interstitial and vacancy point defects in Cz Si. The use of quench cooling in Cz Si pulling is thus well established.

(Emphasis added).

The Examiner randomly cited many portions remotely located and independently described in <u>Wolf et al.</u> However, it is not clear how each portion is relevant to the present invention.

Regarding page 11 of <u>Wolf et al</u>, the oxide precipitate nuclei formed according to the present invention are not interstitial agglomeration defects or agglomerated interstitial point intrinsic defects in dislocation loops, which are disclosed on page 11 of <u>Wolf et al</u>. The quenching from 1421 °C to about 950 °C is different from the cooling of the present invention. Regarding page 49 of <u>Wolf et al</u>, the diffusion (i.e. mobility) of supersaturated Si self-interstitial point defects between about 400 to 1200 °C teaches or suggests nothing about oxide precipitate nuclei. Also, the disclosure on pages 59-61 of <u>Wolf et al</u>. generally discusses oxygen in silicon but does not disclose specific steps for forming oxygen precipitates as recited in the present claims. It is not clear how the nitrogen doping on page 32 of <u>Wolf et al</u> discussed on warpage

resistance is relevant to the method of the present invention. Also, it is not clear how the use of IG (intrinsic gettering) of the substrates prior to deposition on page 141 of Wolf et al is relevant to the recitation of the present claims. It is not clear what is meant by the allegation that the processing parameters are result effective as disclosed on page 61.

Nevertheless, the Examiner urged: "It would have been obvious . . . to combine Iida et al. with Wolf et al. because Iida et al. discloses the **quenching** of Si ingots pulled with the Cz method to **prevent the agglomeration** of vacancy point defects in a certain temperature range and Wolf et al. discloses that the quenching in the temperature range of 1421 to 400 °C prevents agglomeration of interstitial and vacancy point defects in Cz Si. The use of quench cooling in Cz Si pulling is thus well established."

The Examiner bears the initial burden of factually supporting any prima facie conclusion of obviousness, but the Office Action has not established a prima facie case of obviousness. Regarding a prima facie case of obviousness, MPEP states as follows:

> To establish a *prima facie* case of obviousness, three basic criteria must be met. First, there must be some suggestion or motivation, either in the references themselves or in the knowledge generally available to one of ordinary skill in the art, to modify the reference or to combine reference teachings. Second, there must be a reasonable expectation of success. Finally, the prior art reference (or references when combined) must teach or suggest all the claim limitations. The teaching or suggestion to make the claimed combination and the reasonable expectation of success must both be found in the prior art, and not based on applicant's disclosure. *In re Vaeck*, 947 F.2d 488, 20 USPQ2d 1438 (Fed. Cir. 1991). See MPEP § 2143 - § 2143.03 for decisions pertinent to each of these criteria.

In a more logical order, (1) the prior art reference (or references when combined) must teach or suggest **all the claim limitations**; (2) there must be some **suggestion or motivation**

either in the references themselves or in the knowledge generally available to one of ordinary skill in the art, to modify the reference or to combine reference teachings; and (3) there must be a **reasonable expectation of success**.

First, Iida et al (EP0962557) and Wolf et al do not teach or suggest all the claim limitations.  None of the above cited portions, however, specifically discusses "cooling the silicon single crystal with **a cooling rate of not more than 1.2°C/min in the single crystal temperature range of 1000-850°C**, wherein the single crystal has **an oxygen concentration of not less than $12\times10^{17}$ atoms/cm$^3$** (ASTM '79 value)," which are recited in claims 8, 9, 13 and 14.  These recitations are not disclosed in Iida et al., either.

Wolf et al discusses **quenching in the temperature range of 1421 to 400**°C.  Nothing in Wolf et al indicates that the silicon single crystal is cooled with a cooling rate of not more than 1.2°C/min in the single crystal temperature range of 1000-850°C.

Moreover, there is no suggestion or motivation either in the references themselves or in the knowledge generally available to one of ordinary skill in the art, to modify the reference or to combine reference teachings.  To support the conclusion that the claimed invention is directed to obvious subject matter, either the references must expressly or impliedly suggest the claimed invention or the Examiner must present a convincing line of reasoning as to why the artisan would have found the claimed invention to have been obvious in light of the teachings of the references.  *Ex parte Clapp*, 227 USPQ 972, 973 (Bd. Pat. App. & Inter. 1985).  When the motivation to combine the teachings of the references is not immediately apparent, it is the duty of the Examiner to explain why the combination of the teachings is proper. *Ex parte Skinner*, 2 USPQ2d 1788 (Bd. Pat. App. & Inter. 1986).

The Office Action merely lists the portions which the Examiner allegedly thinks relevant. Mere listing of descriptions of the cited reference, however, does not explain why the artisan would have found the claimed invention to have been obvious in light of the teachings of the references.

Also, there cannot be a reasonable expectation of success where the prior art references combined do not teach or suggest all the claim limitations and where there is no suggestion or motivation to modify the reference or to combine reference teachings.

For at least these reasons, claims 8, 9, 13 and 14 patentably distinguish over Iida et al and Wolf et al.

3. **Claims 1, 2, 4 And 5 Are Not Obvious Over Iida et al (EP0909840) In View Of Wolf et al.**

Claims 1, 2, 4 and 5 recite "a cooling rate of not less than 7.3 °C/min in the single crystal temperature range of 1200-1050°C." Iida et al does not teach or suggest "a cooling rate of not less than 7.3 °C/min in the single crystal temperature range of 1200-1050°C." Example 1 of the present application shows sample 1 made by a cooling rate 2.5°C/mm in the range of 1200-1050°C, which satisfies the cooling rate of Iida et al, but the sample 1 exhibits poor oxygen precipitates. Thus, the difference in the cooling rate results in a significant difference which a person of ordinary skill in the art would not have expected from the disclosure of Iida et al. Intrinsic gettering achieved by claims 1, 2, 4 and 5 is not obtained using the cooling rate disclosed by Iida et al.

In response to Applicants' response, the Examiner alleged:

The argument that the combination does not suggest the epitaxial IG wafers produced by the present method is not convincing. The examiner points to the known use of temperature and cooling control to effect both the limitation of defect formation and the promotion of beneficial defect formation (i.e. the oxygen precipitates vital to Intrinsic Gettering). Wolf et al describes IG as beneficial, **this at least suggests a sufficient amount of oxygen precipitates was previously obtained.**

(Emphasis added).

This, however, is not supported by <u>Wolf et al</u>. <u>Wolf et al</u> does not discuss Intrinsic Gettering at page 11 at all. <u>Wolf et al</u> merely describes at the cited portion that "If the pull-rate is high enough, the solidified region cools with sufficient rapidity that the excess point defects are immobilized (or quenched) in the lattice." Although <u>Wolf et al</u> discusses Intrinsic Gettering at pages 66-69, the portion discusses gettering in general. <u>Wolf et al</u> does not indicate Intrinsic Gettering as beneficial in connection with the description at page 11. Nor does it indicate how the beneficial results are obtained by Intrinsic Gettering.

The Examiner also alleged in the response to Applicants' response as follows:

The teach away argument is not convincing. Iida (EP 0990840) discloses few crystal defects without specifying the oxygen concentration required. Iida merely states a preference for "low oxygen to add to the defect avoidance this is [sic] **not rule out higher concentrations**. Page 67 of Wolf et al. suggests optimization of oxygen concentrations for Si wafers.

(Emphasis added).

In <u>Wolf et al</u>, however, there is no disclosure of an oxygen concentration of not less than $12 \times 10^{17}$ atoms/cm$^3$. Nothing indicates that such an oxygen concentration is preferable. There is no suggestion or motivation for a person of ordinary skill in the art to choose such a higher oxygen concentration.

The Examiner further alleged as follows:

The argument that neither Iida [n]or Wolf suggest a method of increasing the density of oxygen precipitates is not convincing. **Wolf et al. discloses oxygen precipitate creation on page 61. This suggests an increase in density. Page 67 discloses a method of nucleation and growth of oxygen precipitates.**

(Emphasis added).

Such general disclosures of <u>Wolf et al</u> do not teach or suggest modifying the disclosure of <u>Iida et al</u> in a way to result in the current invention. Like already discussed, <u>Wolf et al</u> at page 61 does not discuss the oxygen precipitate creation in connection with the description at page 11.

Independent claims 1, 2, 4, and 5 (also claims 8-10, and 13-15) of the present invention recite "wherein the single crystal has an oxygen concentration of not less than $12 \times 10^{17}$ atoms/cm$^3$ (ASTM '79 value)."

In <u>Iida et al</u>, paragraph [0013] states that "a concentration of oxygen in the single crystal ingot is preferably controlled to be $1.0 \times 10^{18}$ atoms/cm$^3$ (ASTM '79 value) or less." In the examples disclosed in <u>Iida et al</u>, oxygen concentration in the single crystal is controlled at $0.8 \times 10^{18}$ atoms/cm$^3$. Thus, <u>Iida et al</u> teaches away from the recitation "wherein the single crystal has an oxygen concentration of not less than $12 \times 10^{17}$ atoms/cm$^3$ (ASTM '79 value)," as recited in the amended claims. The Examiner apparently tries to make the expression "preferably controlled to be $1.0 \times 10^{18}$ atoms/cm$^3$ (ASTM '79 value) or less" as the bases of disclosure of "not less than $12 \times 10^{17}$ atoms/cm$^3$." <u>Iida et al</u>, however, not only is silent about "not less than $12 \times 10^{17}$ atoms/cm$^3$" but rather teaches away the range.

The present invention increases the density of oxide precipitate in the single crystal by promoting formation and growth of thermally stable oxide precipitate nuclei. Therefore, the present invention is not directed to controlling the pulling rate in order to prevent agglomeration

of interstitial and vacancy point defects, which is disclosed. Neither Iida et al nor Wolf et al teach or suggest a process for increasing the density of oxide precipitate in the single crystal.

According to the present method of manufacturing epitaxial wafers, the thermal stability of oxide precipitates is retained even after the epitaxial step without requiring any additional heat treatment step following silicon single crystal pulling up. Thus epitaxial wafers capable of exhibiting high IG (intrinsic gettering) capability are obtained.

Also, the method of manufacturing epitaxial wafers of the present invention provides epitaxial wafers allowing formation of a sufficient amount of oxide precipitates upon high temperature heat treatment in the device while preventing the generation of epitaxial layer defects even when they are manufactured from a silicon single crystal grown with nitrogen doping.

For at least these reasons, claims 1, 2, 4, and 5 patentably distinguish over Iida et al.

## 4. Claims 10 And 15 Are Not Obvious Over Iida et al (EP0909840) In View Of Wolf et al.

Claims 10 and 15 are directed to a method of manufacturing a silicon single crystal doped with $5 \times 10^{13}$ atoms/cm$^3$ to $1 \times 10^{16}$ atoms/cm$^3$ of nitrogen. These claims recite "a cooling rate of not less than 6.5°C/min in the single crystal temperature range of 1150-800°C" and "wherein the single crystal has an oxygen concentration of not less than $12 \times 10^{17}$ atoms/cm$^3$ (ASTM '79 value)."

These recitations are not taught or suggested by the cited references. If the single crystal is not cooled with this rapid cooling, too many thermally stable oxide precipitates are formed in the single crystal resulting in epitaxial defects in the epitaxial growth process.

For at least these reasons, claims 10 and 15 patentably distinguish over <u>Iida et al</u> and <u>Wolf et al.</u>  Claim 11, depending from claims 8-10, and claim 16, depending from claims 13-15, also patentably distinguish over <u>Iida et al</u> and <u>Wolf et al</u> for at least the same reasons.
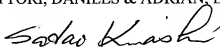
## X. CONCLUSION

For the foregoing reasons, the Honorable Board of Patent Appeals and Interferences is respectfully requested to reverse the 35 USC §103(a) rejection of claims 1, 2, 4, 5, 8-11 and 13-16 on appeal.

In the event this paper is not timely filed, appellant hereby petitions for an appropriate extension of time. The fee for any such extension may be charged to our Deposit Account No. 50-2866, along with any other additional fees which may be required with respect to this paper.

Respectfully submitted,

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Enclosures: Appendix containing Claims on Appeal

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**CLAIMS ON APPEAL**

1.   A method of producing silicon single crystals which comprises:

pulling up the silicon single crystal in the Czochralski method, with a cooling rate of not less than 7.3 °C/min in the single crystal temperature range of 1200-1050°C,

wherein the single crystal has an oxygen concentration of not less than $12 \times 10^{17}$ atoms/cm$^3$ (ASTM '79 value), and the single crystal is not nitrogen doped.

2.   A method of producing silicon single crystals which comprises:

pulling up the silicon single crystal in the Czochralski method, with a cooling rate of not less than 7.3°C/min in the single crystal temperature range of 1200-1050°C; and then

cooling the single crystal at a cooling rate of not more than 3.5°C/min in the single crystal temperature range of 1000-700°C,

wherein the single crystal has an oxygen concentration of not less than $12 \times 10^{17}$ atoms/cm$^3$ (ASTM '79 value), and the single crystal is not nitrogen doped.

3.   (Canceled).

4.   A method of manufacturing epitaxial wafers which comprises:

forming an epitaxial layer on the surface of a silicon wafer sliced from a silicon single crystal produced by the Czochralski method with a cooling rate of not less than 7.3°C/min in the single crystal temperature range of 1200-1050°C,

wherein the single crystal has an oxygen concentration of not less than $12 \times 10^{17}$ atoms/cm$^3$ (ASTM '79 value), and the single crystal is not nitrogen doped.

5.  A method of manufacturing epitaxial wafers which comprises:

forming an epitaxial layer to grow on the surface of a silicon wafer sliced from a silicon single crystal produced by the Czochralski method with a cooling rate of not less than 7.3°C/min in the single crystal temperature range of 1200-1050°C; and then

cooling the single crystal at a cooling rate of not more than 3.5°C/min in the single crystal temperature range of 1000-700°C,

wherein the single crystal has an oxygen concentration of not less than $12 \times 10^{17}$ atoms/cm$^3$ (ASTM '79 value), and the single crystal is not nitrogen doped.

6-7.  (Canceled).

8.  A method of producing silicon single crystals which comprises:

pulling up a silicon single crystal doped with $1 \times 10^{12}$ atoms/cm$^3$ to $1 \times 10^{14}$ atoms/cm$^3$ of nitrogen in the Czochralski method;

cooling the silicon single crystal with a cooling rate of not more than 1.2°C/min in the single crystal temperature range of 1000-850°C,

wherein the single crystal has an oxygen concentration of not less than $12 \times 10^{17}$ atoms/cm$^3$ (ASTM '79 value).

9. A method of producing silicon single crystals which comprises:

pulling up a silicon single crystal doped with $1 \times 10^{12}$ atoms/cm$^3$ to $1 \times 10^{14}$ atoms/cm$^3$ of nitrogen in the Czochralski method, with a cooling rate of not less than 2.7°C/min in the single crystal temperature range of 1150-1020°C; and then

cooling the silicon single crystal at a cooling rate of not more than 1.2°C/min in the single crystal temperature range of 1000-850°C,

wherein the single crystal has an oxygen concentration of not less than $12 \times 10^{17}$ atoms/cm$^3$ (ASTM '79 value).

10. A method of producing silicon single crystals which comprises:

pulling up a silicon single crystal doped with $5 \times 10^{13}$ atoms/cm$^3$ to $1 \times 10^{16}$ atoms/cm$^3$ of nitrogen in the Czochralski method, with a cooling rate of not less than 6.5°C/min in the single crystal temperature range of 1150-800°C,

wherein the single crystal has an oxygen concentration of not less than $12 \times 10^{17}$ atoms/cm$^3$ (ASTM '79 value).

11. A method of producing silicon single crystals as claimed in any of Claims 8 to 10, wherein the single crystal has an oxygen concentration of not less than $4 \times 10^{17}$ atoms/cm$^3$ (ASTM '79).

12. (Canceled)

13. A method of manufacturing epitaxial wafers which comprises:

forming an epitaxial layer to grow on the surface of a silicon wafer sliced from a silicon single crystal doped with $1 \times 10^{12}$ atoms/cm$^3$ to $1 \times 10^{14}$ atoms/cm$^3$ of nitrogen as produced by the Czochralski method; and then

cooling the epitaxial layer with a cooling rate of not more than 1.2°C/min in the single crystal temperature range of 1000-850°C,

wherein the single crystal has an oxygen concentration of not less than $12 \times 10^{17}$ atoms/cm$^3$ (ASTM '79 value).

14. A method of manufacturing epitaxial wafers which comprises:

forming an epitaxial layer on the surface of a silicon wafer sliced from a silicon single crystal doped with $1 \times 10^{12}$ atoms/cm$^3$ to $1 \times 10^{14}$ atoms/cm$^3$ of nitrogen as produced by the Czochralski method by with a cooling rate of not less than 2.7°C/min in the single crystal temperature range of 1150-1020°C; and then

cooling the epitaxial layer at a cooling rate of not more than 1.2°C/min in the single crystal temperature range of 1000-850°C,

wherein the single crystal has an oxygen concentration of not less than $12 \times 10^{17}$ atoms/cm$^3$ (ASTM '79 value).

15. A method of manufacturing epitaxial wafers which comprises:

forming an epitaxial layer on the surface of a silicon wafer sliced from a silicon single crystal doped with $5 \times 10^{13}$ atoms/cm$^3$ to $1 \times 10^{16}$ atoms/cm$^3$ of nitrogen as produced by the

Czochralski method with a cooling rate of not less than 6.5°C/min in the crystal temperature range of 1150-800°C,

wherein the single crystal has an oxygen concentration of not less than $12 \times 10^{17}$ atoms/cm$^3$ (ASTM '79 value).


16.    A method of manufacturing epitaxial wafers as claimed in any of Claims 13 to 15, wherein the silicon wafer sliced out has an oxygen concentration of not less than $4 \times 10^{17}$ atoms/cm$^3$ (ASTM '79).